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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

U.S. Patent

Application of: M. NAKAMURA ET AL

Serial Number : 09/998,764

Filed : November 29, 2001

For : PIGMENT DISPERSION LIQUID, MANUFACTURING  
PROCESS THEREOF, PIGMENT INK FOR INK JETTING,  
AND INK JET IMAGE RECORDING METHOD EMPLOYING  
THE SAME

Group Art Unit: 1755

Examiner : SHALIE A. MANLOVE

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DECLARATION UNDER 37 C.F.R. 1.132

Hon. Commissioner of Patents  
and Trademarks  
Washington, D.C. 20231

Sir:

I, MASAKI NAKAMURA, hereby declare and say as follows:

That I am a post graduate from Chiba University having been awarded a Masters Degree in Organic Synthetic Chemistry in March 1982.

That since April 1982, I have been employed by Konica Corporation, the owner of the above-identified application. During my employment, I have been engaged in the research and the study of photographic materials and ink jet recording materials in the Research and Development Laboratory of my company.

That I am a co-inventor of the present application.

That I am familiar with the subject matter of the present invention.

What follows is an accurate summary of experiments conducted according to my detailed instructions and under my personal supervision, and the results obtained therefrom.

#### Comparative tests

1. Claims 1-8 are rejected under 35 U.S.C. 102(b) as being anticipated by Scaringe et al. (US 6132501). Claims 1-8 are rejected under 35 U.S.C. 102(b) as being anticipated by Bishop et al. (EP 0776952). However, neither Scaringe et al. nor Bishop et al. disclose a pigment dispersion liquid containing a water soluble polymer, much less a water soluble polymer with a weight average molecular weight of 1000 to 200,000, as recited in instant claim. Therefore, instant claims are not anticipated by, nor obvious, over the cited references. In spite of the above, in order to show unexpected results of the invention, additional comparative tests were carried out.

#### 2. Preparation of Pigment dispersion liquids

Preparation of Pigment dispersion liquid 101 (Comparative)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl pyrrolidone (PVP1) having a weight average molecular weight of 900 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 101 was obtained. Pigment dispersion liquid 101 was prepared in the same manner as in Pigment dispersion

liquid 1 of Example 1-1 of the Specification, except that Solution 101 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 102 (Inventive)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl pyrrolidone (PVP2) having a weight average molecular weight of 15,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 102 was obtained. Pigment dispersion liquid 102 was prepared in the same manner as in Pigment dispersion liquid 1 of Example 1-1 of the Specification, except that Solution 102 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 103 (Inventive)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl pyrrolidone (PVP3) having a weight average molecular weight of 80,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 103 was obtained. Pigment dispersion liquid 103 was prepared in the same manner as in Pigment dispersion liquid 1 of Example 1-1 of the Specification, except that Solution 103 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 104 (Inventive)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl pyrrolidone (PVP4) having a weight average molecular weight of 180,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 104 was obtained. Pigment dispersion liquid 104 was prepared in the same manner as in Pigment dispersion

liquid 1 of Example 1-1 of the Specification, except that Solution 104 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 105 (Comparative)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl pyrrolidone (PVP5) having a weight average molecular weight of 210,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 105 was obtained. Pigment dispersion liquid 105 was prepared in the same manner as in Pigment dispersion liquid 1 of Example 1-1 of the Specification, except that Solution 105 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 201 (Comparative)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl alcohol (PVA1) having a weight average molecular weight of 900 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 201 was obtained. Pigment dispersion liquid 201 was prepared in the same manner as in Pigment dispersion liquid 1 of Example 1-1 of the Specification, except that Solution 201 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 202 (Inventive)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl alcohol (PVA2) having a weight average molecular weight of 15,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 202 was obtained. Pigment dispersion liquid 202 was prepared in the same manner as in Pigment dispersion

liquid 1 of Example 1-1 of the Specification, except that Solution 202 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 203 (Inventive)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl alcohol (PVA3) having a weight average molecular weight of 80,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 203 was obtained. Pigment dispersion liquid 203 was prepared in the same manner as in Pigment dispersion liquid 1 of Example 1-1 of the Specification, except that Solution 203 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 204 (Inventive)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl alcohol (PVA4) having a weight average molecular weight of 180,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 204 was obtained. Pigment dispersion liquid 204 was prepared in the same manner as in Pigment dispersion liquid 1 of Example 1-1 of the Specification, except that Solution 204 was used instead of Solution A1.

Preparation of Pigment dispersion liquid 205 (Comparative)

C.I. Pigment Red 122 of 50 g and 100 g of polyvinyl alcohol (PVA5) having a weight average molecular weight of 210,000 were dissolved in a mixture solvent of 750 g of DMSO (dimethylsulfoxide) and 250 g of an aqueous 10% sodium hydroxide solution, and filtered through a Millipore filter paper of a 1  $\mu$ m pore diameter. Thus, Solution 205 was obtained. Pigment dispersion liquid 205 was prepared in the same manner as in Pigment dispersion

liquid 1 of Example 1-1 of the Specification, except that Solution 205 was used instead of Solution A1.

With respect to the pigment dispersion liquids obtained above, the average primary particle size of dispersed particles and  $D_{10}$ ,  $D_{50}$ , and  $D_{90}$  were measured in the same manner as in Example 1-1 of the present Specification. The results are shown in Table 101.

Table 101

Pigment dispersion liquid No.	Water soluble polymer	Weight average molecular weight	Average primary particle size (nm)	$D_{10}$	$D_{50}$	$D_{90}-D_{10}$	Remarks
101	PVP1	900	20	7	39	32	Comp.
102	PVP2	15,000	13	5	21	16	Inv.
103	PVP3	80,000	14	6	24	18	Inv.
104	PVP4	180,000	18	6	28	22	Inv.
105	PVP5	210,000	24	6	30	24	Comp.
201	PVA1	900	23	6	40	34	Comp.
202	PVA2	15,000	10	4	15	11	Inv.
203	PVA3	80,000	13	5	20	15	Inv.
204	PVA4	180,000	17	5	27	22	Inv.
205	PVA5	210,000	29	6	29	23	Comp.

Inv.: Inventive, Comp. Comparative

### 3. Preparation of Pigment inks

Pigment inks 101 through 105 were prepared from pigment dispersion liquids 101 through 105, respectively, in the same manner as in Example 1-2 of the present Specification. Pigment inks 201 through 205 were prepared from pigment dispersion liquids 201 through 205, respectively, in the same manner as in Example 1-2 of the present Specification.

#### 4. Evaluation of Pigment inks

The resulting pigment inks obtained above were evaluated for dispersion stability, ink recording head nozzle clogging, bronzing and glossiness in the same manner as in Example 1-2 of the present Specification. The results are shown in Table 102.

Table 102

Pigment ink No.	Pigment dispersion liquid No. used in pigment inks	Dispersion stability	Nozzle clogging	Plain paper sheet		Porous paper sheet		Remarks
				Bronzing	Glossiness	Bronzing	Glossiness	
101	101	B	B	C	C	C	C	Comp.
102	102	A	A	B	B	A	A	Inv.
103	103	A	A	B	B	A	A	Inv.
104	104	A	B	B	B	A	A	Inv.
105	105	A	D	C	D	B	D	Comp.
201	201	B	B	C	D	C	C	Comp.
202	202	A	A	B	B	A	A	Inv.
203	203	A	A	B	B	A	A	Inv.
204	204	A	B	B	B	A	A	Inv.
205	205	A	D	B	D	B	D	Comp.

Inv.: Inventive, Comp. Comparative

As is apparent from Tables 101 and 102 above, inventive pigment dispersion liquids comprise pigment particles falling within the claimed scope of (D<sub>90</sub> -D<sub>10</sub>). Further, inventive pigment inks, comprising the inventive pigment dispersion liquids, provide good dispersion stability, good resistance to nozzle clogging during ink jetting, and images with minimized bronzing and good glossiness, as compared with comparative pigment inks, comprising comparative pigment dispersion liquids. These

results are unexpected to one of ordinary skill in the art. In view of the above, it would not have been obvious to one of ordinary skill in the art to attain the invention over Scaringe et al., Bishop et al., or a combination thereof. Accordingly, we believe that instant claim 1, and all the claims, which depend therefrom, are in a situation of allowability.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001, of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: June 26, 2003

Masaki Nakamura

MASAKI NAKAMURA